Nitrogen in Fertilizer

LECO Corporation; Saint Joseph, Michigan USA

Instrument: TruMac® N

Introduction

Nitrogen is one of the most important elements for plant development and is the macronutrient that is most often found to be deficient in arable soils used for crop production. Nitrogen plays a key role in promoting lush vigorous growth and development of plants that often leads to an increase in the yield from the plant. Fertilizers are utilized to re-introduce nitrogen back into arable soils. Fertilizers can be grouped by their makeup and/or origins into categories including inorganic and/or synthetic (including nitrates, ammonium, and urea) and organic (including compost materials and manures). The accurate and precise determination of nitrogen in all fertilizer types is not only important in the process of blending and preparing the fertilizer material for use but also will have significant impact on the commercial value and guarantee of the fertilizer.

The LECO TruMac N combustion nitrogen determinator has been designed to easily handle the fertilizer sample's mass in addition to the mass of the required sucrose addition (AOAC 993.13), while maintaining a rapid analysis time with automatic post-analysis ash removal.

The LECO TruMac N is a macro combustion nitrogen determinator that utilizes a pure oxygen environment in a ceramic horizontal furnace operating at a high temperature (1350°C), with large ceramic boats for the macro sample combustion process. A combustion gas collection and handling system using helium carrier gas and a thermal conductivity cell for the detection of nitrogen are also utilized.

Method Reference

AOAC 993.13

Sample Preparation

Samples must be of uniform consistency to produce suitable results

Accessories

528-203 Crucibles, 502-343 Nickel Boat Liners, 501-441 Sucrose

Calibration Samples

Ammonium Nitrate, Ammonium Sulfate, NIST SRM 913 Uric Acid, NIST SRM 194 Ammonium Dihydrogen Phosphate, Urea, or other suitable reference materials

*Analysis Parameters**

Furnace Temperature	1350°C
TE Cooler Temperature	5°C
Dehydration Time	0 seconds
Purge Cycles	2 seconds

Element Parameters

Baseline Delay Time	6 seconds
Minimum Analysis Time	35 seconds
Comparator Level	100.00
Endline Time	2 seconds
Conversion Factor	1.00
Significant Digits	5
TC Baseline Time	10 seconds

Burn Profile

Burn Cycle	Lance Flow	Purge Flow	Time
1	Off	On	5 seconds
2	On	On	5 seconds
3	On	Off	END

Ballast Parameters

Equilibrate Time	30 seconds
Not Filled Timeout	300 seconds

Aliquot Loop

Equilibrate Pressure Time	4 seconds
High Precision	Yes
High Speed	No

**Refer to TruMac Operator's Instruction Manual for Method Parameter definitions.*

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Condition the system by analyzing 3-5 blanks (crucible is not required).
3. Determine blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as the sample name.
   b. Add ~0.4 g sucrose to a 528-203 Crucible.
   c. Transfer crucible to the appropriate position of the autoloader.
   d. Repeat steps 3a through 3c a minimum of three times.
   e. Initiate the analysis sequence (F5).
   f. Set the blank following the procedure outlined in the operator's instruction manual.
4. Calibrate.
   a. Weigh ~0.1 g of Ammonium Nitrate or other suitable calibration sample into a 528-203 Crucible; enter mass and sample identification into Sample Login (F3).
   b. Add ~0.4 g of sucrose to sample and mix thoroughly.
   c. Transfer crucible to the appropriate position of the autoloader.
   d. Repeat steps 4a through 4c a minimum of three times.

Organic Application Note

e. Initiate the analysis sequence (F5).
f. Calibrate the instrument following the procedure outlined in the operator's instruction manual.

Note: Multi-point (fractional weight or multiple calibration samples) may be used to calibrate if desired. A TruMac can be calibrated using several replicates of a single mass range (0.1 g) of Ammonium Nitrate utilizing a single standard calibration. This is a cost-effective and simple process. The calibration can be verified by analyzing different compounds such as Ammonium Sulfate (0.1 g) and/or Urea (0.1 g).

5. Analyze Samples (powder/granular).
   a. Weigh ~0.1 g sample into a 528-203 Crucible; enter mass and sample identification into Sample Login (F3).
   b. Add ~0.4 g of sucrose to sample and mix thoroughly.
   c. Transfer crucible to the appropriate position of the autoloader.
   d. Repeat steps 5a through 5c for each sample to be analyzed.
   e. Initiate the analysis sequence (F5).
6. Analyze Samples (liquid).
   a. Place a 502-343 Nickel Boat Liner into a 528-203 Crucible.
   b. Weigh ~0.1 to 0.15 g of liquid fertilizer into the Nickel Boat Liner; enter mass and sample identification into Sample Login (F3).
   c. Transfer crucible to the appropriate position of the autoloader.
   d. Repeat steps 6a through 6c for each liquid sample to be analyzed.
   e. Initiate the analysis sequence (F5).

Note: Liquid fertilizer does not require a sucrose addition.

Typical Results

Sample	*Mass g*	*% N*
Urea	0.1046	46.71
@ 46.64% N	0.1072	46.56
	0.1003	46.62
	0.1039	46.66
	0.1023	46.70
	X =	***46.65***
	s =	***0.06***
Ammonium	0.1015	34.93
Nitrate	0.0992	35.09
@ 35% N	0.1107	34.97
	0.1043	34.92
	0.1050	34.91
	X =	***34.96***
	s =	***0.07***
Ammonium	0.1145	21.12
Sulfate	0.1014	21.16
@ 21.2% N	0.1039	21.22
	0.1062	21.10
	0.1045	21.20
	X =	***21.16***
	s =	***0.05***
Liquid	0.1244	32.24
Fertilizer	0.1289	32.22
	0.1200	32.28
	0.1150	32.30
	0.1174	32.27
	X =	***32.26***
	s =	***0.03***
Liquid	0.1211	19.48
Fertilizer	0.1170	19.47
	0.1162	19.46
	0.1350	19.42
	0.1270	19.45
	X =	***19.46***
	s =	***0.02***

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